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PRECIPITATES IN A NIOBIUM-ZIRCONIUM-CARBON
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Work performed for
U.S. DEPARTMENT OF ENERGY
Nuclear Energy
Reactor Systems Development and Technology

Prepared for
The Metallurgical Society Fall Meeting
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CHARACTERIZATION OF PRECIPITATES IN A Nb-Zr-C ALLOY

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SUMMARY

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A niobium alloy with 1 percent zirconium and 0.063 percent carbon by weight was investigated in the as-rolled and annealed conditions, and after high-temperature (1350 and 1400 K) exposure with and without an applied stress. In the as-rolled and in the annealed conditions, large metastable carbides were observed in addition to a regular distribution of small particles. During the high-temperature exposure, the majority of the large carbides were dissolved and a more stable carbide phase formed. This finely dispersed phase had a composition determined to be ~70 percent ZrC and ~30 percent NbC and showed some evidence of an orientation relationship with the matrix. The precipitates appeared to coarsen slightly after ~5000 hr exposure at temperatures between 1350 and 1400 K. This same high-temperature exposure in the presence of an applied stress resulted in a decrease in the size and in the interparticle spacing of the stable precipitates. However, the composition of the precipitate phase and its ability to pin dislocations were not affected by the temperature or stress conditions.

INTRODUCTION

Niobium-zirconium-carbon alloys were first developed for space energy conversion applications due to their elevated temperature creep resistance, resistance to corrosion by liquid alkali metals, and relatively low density. These alloys rely on complex carbide precipitates for strengthening rather than zirconium oxide precipitation as in binary niobium-1 percent zirconium. It has been established that carbide precipitates are more effective strengtheners, but the actual composition, morphology, and aging characteristics of the second phase in alloys containing carbon have not been fully investigated. Several workers have analyzed precipitation in the Nb-Zr-C system (Arzamozov and Vasil'eva, 1978; Begley et al., 1963; Cuff, 1962; Kissil et al., 1976, 1978; Korotayev et al., 1980, 1981; Lyutiy et al., 1978; Maksimovich et al., 1978; Ostermann and Bollenrath, 1966, Ostermann, 1971; Tsvikilevich, 1980), but better understanding of the kinetics of aging reactions with respect to the intended service conditions is required for determination of design parameters. The present work was undertaken to investigate the carbide morphology in a Nb-Zr-C alloy heat treated with and without the presence of an applied stress.

The strength of Nb-Zr-C alloys has been shown to be much higher than binary Nb-Zr or Nb-C alloys (DelGrosso et al., 1967; Grigorovich and Sheftel, 1982). It is assumed that the suppression of the transition of the carbide phase from coherent to incoherent (as in binary Nb-C alloys) is due to the formation of complexes of carbon, niobium, and zirconium atoms (Korotayev et al., 1981). The increased strength of the ternary alloy is due, therefore, to the precipitation of stable, zirconium-enriched (Nb,Zr)C instead of NbC, which breaks down at lower temperatures than the complex carbide. The increased stability of the (Nb,Zr)C carbide can be attributed to the relative immobility of

zirconium in niobium due to its larger atomic size. In addition, although the precipitate phase may contain oxygen, it must be primarily a carbide due to the increased strengthening of Nb-Zr-C alloys over Nb-1Zr with ZrO₂ precipitates.

MATERIAL

The material used in this study was received from the Oak Ridge National Laboratory in the form of as-rolled, 1-mm thick tensile specimens. This material was from a single arc-melt followed by a single primary breakdown extrusion in the 1350 K regime. It was subsequently processed into tube material, which was split and rolled to produce sheet specimens. All material was annealed for 1 hr at 1755 K then 2 hr at 1475 K prior to any testing. Annealing treatments were conducted in titanium sublimation pumped systems at a pressure below 10⁻⁵ Pa and included wrapping each specimen in cleaned tantalum foil.

Two of the sheet samples were cut into smaller pieces, wrapped in Nb-1Zr foil, and aged at 1350 K with no applied stress. The heat treatments were carried out in LN₂ trapped oil diffusion pumped systems at a pressure of ~1x10⁻⁴ Pa. Several samples were aged in the presence of an applied stress at 1350 and 1400 K as described by Titran (1986) in internally loaded chambers at constant load. The vacuum, maintained by bakeable titanium sublimation pumps, was initially ~10⁻⁵ and ranged to 5x10⁻⁷ Pa after several hundred hours.

EXPERIMENTAL PROCEDURE

Traditional etching for optical and scanning electron metallography was a difficult technique for examining the carbide structure due to the tendency of the particles to fall out of the structure during etching. This was due to the fact that the etchant removed material underneath the particles without attacking the carbides. An anodic staining technique was tried (Crouse, 1965), but was not easily interpretable due to the small size of the precipitates. Limited success was obtained using a nitric/hydrofluoric acid etchant.

Phase extraction was performed on specimens supported with platinum wires in 100 ml of bromine in 900 ml of methanol with 10 g of tartaric acid in solution. The cell was periodically subjected to ultrasound to disperse any passive layer on the surface of the samples. X-ray analysis was used to identify and to determine the composition of the precipitate phase. Chemical analysis was also performed on the extracted material by inductively coupled plasma atomic emission spectrometry.

Transmission electron microscopy foils were cut by electric discharge machining. After the oxide layer was removed by hand polishing, some samples were jet thinned in an electrolyte consisting of 936 ml methanol, 62.8 ml H₂SO₄, 40 ml butyl alcohol, and 1.5 ml HF. The foils were prepared using a Struers Tenupol apparatus operated at a current density of ~27 mA/mm². Although the large carbide particles tended to fall out of the foils during thinning, adequate thin areas were obtained. In addition, some foils were dimpled then ion-milled.

RESULTS AND DISCUSSION

Chemical Analysis

The oxygen contents increased after aging with and without an applied stress as evidenced in table I, which lists the compositions in both weight percent and atomic percent. This increase was expected and realistic since the vacuum levels at which the material was tested approximated what the material would see during use in space power systems. The oxygen content of the alloy was 2.5 times greater after aging without applied stress, and was four times greater after aging with an applied stress of 40 MPa. These two cases were for approximately the same time and temperature, and the vacuum was one to two orders of magnitude better for the sample under stress. The larger increase in this case is attributed to the increased oxygen mobility in the presence of the applied stress. The increased oxygen content, however, was not discernible through metallography or phase extraction, indicating that most of the oxygen remained in solution.

Metallography

The microstructures of the as-rolled condition and the annealed condition for the Nb-Zr-C alloy are shown in figure 1. Various shapes and sizes of particles were noticeable in both microstructures. The morphology ranged from massive 5- to 10- μm particles decorating grain boundaries to submicron needle-like particles. The large particles observed are believed to be primary metastable carbides of Nb_2C formed during the initial solidification which were neither broken up nor dissolved during extrusion or during the sheet rolling process. The annealed material had a mixture of elongated and equiaxed grains with an average grain size of $\sim 25 \mu\text{m}$.

It appears from the size of the carbides in the annealed condition (fig. 1(b)) that the anneal was insufficient to solution the large carbide particles. Aged samples (fig. 2), however, show that these particles are dissolved and that a uniform distribution of a new precipitate forms, with a size ranging from 500 to 1000 nm. All of the samples exposed to high temperatures for time greater than 500 hr exhibited this same pattern of precipitation. Scanning electron microscopy could not differentiate between short and long exposures to temperature and/or stress.

Phase Extraction

Analysis of the phase-extracted material is listed in table II. Material in the as-rolled condition showed mostly Nb_2C and a very minor phase with $a_0 = 0.468 \text{ nm}$, and with virtually no zirconium in the precipitate phase. In the annealed condition, both Nb_2C and an fcc phase with $a_0 = 0.468$ were identified as major phases, though the zirconium content of the precipitate did not increase appreciably. In those samples exposed for times greater than 100 hr, the only phase identified was face centered cubic with a lattice parameter between 0.461 and 0.467 nm. It is interesting to note that Korotayev et al. (1981) identified a phase with a lattice parameter of 0.462 nm using electron diffraction analysis of extracted replicas of Nb-1Zr-0.1C alloy samples

quenched from 2075 K and aged for 100 hr at 1275 K. They state that the transformation to the stable monocarbide is largely completed in 100 hr at temperatures over 1300 K. Khandarov et al. (1978), however, estimated that full decomposition of the metastable carbide would require aging over 10 000 hr at 1375 K.

Although there is considerable scatter in the data, the zirconium content of the precipitate phase was virtually constant after ~300 hr at either 1350 or 1400 K. A wide variety of precipitate compositions are possible, some of which are listed in table III. It is possible that the extracted material is a complex phase consisting of all of these compounds, but it is more likely that it is primarily a mixture of ZrC and NbC, the stable carbide phases. If we assume that these are the components of the precipitate, it is possible to determine the relative amounts of each carbide present by referring to figure 3, which plots the solubility relationship between NbC and ZrC (Norton and Mowry, 1949). If the precipitate phase is in fact (Zr,Nb)C with a lattice parameter of 0.462 nm, the composition is 70 percent ZrC and 30 percent NbC, a Zr/Nb ratio of ~2.3. The Zr/Nb ratio found from the chemical analysis and the lattice parameter values from x-ray diffraction lead to the conclusion that the composition of the precipitate is very close to the stoichiometric monocarbide.

Considering the increase of the oxygen content during aging, it is likely that oxygen is present in at least some of the precipitate particles. McCoy and Douglas (1961) predict that when oxygen, nitrogen, and carbon are all present in solution, ZrO_2 will form preferentially since both free energy of formation values and diffusion rates favor the formation of the oxide over the nitride or carbide. Some experimental support of this was provided by Korotayev et al. (1980), who observed an internal oxidation front in a Nb-1Zr-0.1C alloy where the original carbide phase transformed to ZrO_2 platelets. No ZrO_2 was observed in the x-ray diffraction analysis, but since the lattice parameter of ZrO (a metastable phase which does not readily form on its own) is so close to that of ZrC and NbC, it is impossible to tell from this analysis if the oxygen absorbed during aging stays in solution or forms the complex (Nb,Zr)(O,C). Since virtually no nitrogen absorption was detected, we do not expect nitride formation.

There is some evidence that internal oxidation is a bulk rather than a surface phenomenon when the sample is under an applied stress, attributed to increased oxygen mobility (Korotayev et al., 1980; Lyutiy et al., 1984). For the carbides, however, Tsvikilevich (1980) observed that the application of a load during aging at 1166 and 1366 K did not change the phase composition and form of the carbide precipitates. Ostermann (1971) points out that the large range of lattice dimensions when the MC_{1-x} carbide deviates from stoichiometry offers the likely possibility of perfect atomic matching across the habit plane. In addition, Lyutiy et al. (1978) found virtually no difference in the size or interparticle spacing of the precipitates with and without an applied stress of ~15 MPa in samples exposed for up to 10 000 hr at 1375 K, although they do observe increased strength in those samples aged in the presence of the applied stress. Wilcox and Allen (1967) also finds some evidence of dynamic strengthening during creep of a Nb-W-Zr alloy.

Transmission Electron Microscopy

Electron microscopy of a sample aged for 25 hr at 1350 K typically revealed flat precipitates in three perpendicular variants, approximately 100 to 150 nm in size, spaced from 100 to 200 nm apart. They were imaged using the spots pointed out by the arrow in figure 4. The lattice parameter of this phase could not be identified by the diffracted spot spacing, but the spots indicate an orientation relationship with the matrix.

In samples aged in the presence of a stress, similar precipitates, approximately 50 to 100 nm in size, were found which were generally closely and regularly spaced at ~50 to 100 nm. These were tentatively identified as $(\text{Zr,Nb})\text{C}$ precipitates with a lattice parameter of 0.462 nm by the diffraction spot spacing. The images in figures 5 and 6 are of a sample which was under an applied stress for 650 hr at 1400 K. The precipitates were imaged in dark field electron microscopy using the $(200)_{\text{Carbide}}$ and $(111)_{\text{Carbide}}$ spots, respectively. The square shape of the precipitates indicates that little or no coarsening has taken place in this time.

In samples aged in the presence of an applied stress for longer times, the precipitates grew in size from 100 to 150 nm across and took on a more rounded shape, indicating some coarsening had occurred. The sample shown in figure 7 was under an applied stress for 4770 hrs at 1350 K. The Moire fringes apparent in the precipitate indicate a lattice mismatch. All of these particles appeared capable of pinning dislocations to at least some extent, and very large dislocation tangles were common. Suggestions of a dislocation cell structure were observed, but the large amount of small particles appeared to hamper any dislocation alignment.

Lyutyi et al. (1978) found an increase in the average size of the particles from 86 to 133 nm upon aging for 5000 hr at 1375 K. The interparticle spacing also increased from 390 to 450 nm. Exposure at this temperature for 10 000 hr resulted in a particle size of 150 nm and an interparticle spacing of 450 nm. Lyutyi et al. reported virtually no difference when a stress of 15 MPa was applied. This particle growth appears to be in accord with that found in this Nb-Zr-C alloy within experimental scatter.

CONCLUDING REMARKS

The initial annealing treatment (1 hr at 1755 K + 2 hr at 1475 K), intended to dissolve the large metastable carbides, was not sufficient. However, these particles did dissolve during aging at 1350 and at 1400 K and a fine, more stable precipitate formed. It is believed that some of these finely dispersed particles are $(\text{Zr,Nb})\text{C}$ with a Zr/Nb ratio of ~2.3. It is also possible that these precipitates contain some oxygen. The composition of the particles did not change with time at temperature, and all of the particles observed appeared to be effective at pinning dislocations.

Aging with and without the presence of an applied stress appears to cause precipitates to form with some evidence of an orientation relationship with the matrix. Square precipitates of $(\text{Zr,Nb})\text{C}$ form which show little evidence of coarsening at 1400 K for 650 hr, but appear to grow to approximately twice their original size after 5000 hr at 1350 K. Precipitates aged in the presence

of a stress exhibited a somewhat smaller size and interparticle spacing than those aged without an applied stress. The increase in the diffusion rate of carbon, oxygen, and zirconium in the presence of a stress may be the reason for this. However, the particles were very similar to those observed in a sample aged at 1350 K for only 25 hr with no applied stress, indicating the long-term stability of these precipitates. In conclusion, the fine (Zr,Nb)C precipitate is a very stable particle and, as such, is an effective creep strengthener in the 1350 K regime.

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TABLE I. - CHEMICAL ANALYSIS OF Nb-Zr-C ALLOY

	As-rolled		Aged at 1350 K for ~1500 hr			
			No stress		Stress = 40 MPa	
	At %	Wt %	At %	Wt %	At %	Wt %
Zirconium	0.90	0.90	0.90	0.90	0.90	0.90
Carbon	.49	.063	.49	.064	.49	.063
Oxygen	.046	.008	.110	.019	.184	.031
Nitrogen	.035	.005	.013	.002	.040	.006
Niobium	balance		balance		balance	

TABLE II. - ANALYSIS OF PHASE-EXTRACTED MATERIAL

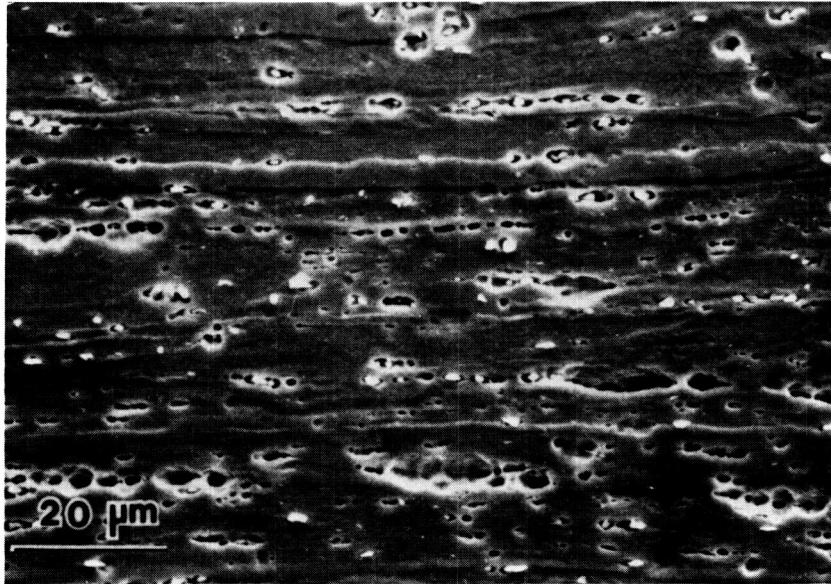
Condition	Aging conditions					Total strain, percent	Total hr at temperature	Zr/Nb ratio	a ₀ , nm
	No stress, hr	Temperature, K	Stress		Temperature, K				
			hr	Mpa					
As-rolled	0	----	----	--	----	---	0	0.01	Nb ₂ C+ .468
Annealed	3	1350	----	--	----	---	3	.04	Nb ₂ C+ .458
↓	100	1350	----	--	----	---	100	.27	.461
	315	1350	----	--	----	---	315	1.62	.462
	1000	1400	329	35	1400	1.8	1329	2.30	.462
	1000	1400	246	40	1350	1.4	1246	2.41	.462
	1246	(a)	----	--	----	---	1246	1.87	.463
	1000	1350	531	40	1350	1.7	1531	2.11	.461
	1531	1350	----	--	----	---	1531	2.39	.463
	1296	1350	1296	35	1400	1.9	1296	1.11	.465
	1296	1350	1489	45	1350	2.2	1489	1.58	.467
	1489	1350	----	--	----	---	1489	1.31	.462
	1489	1350	2929	28	1400	1.2	2929	2.42	.465
	↓	----	----	----	--	----	---	----	----

^a1000 hr at 1400 K + 246 hr at 1350 K.

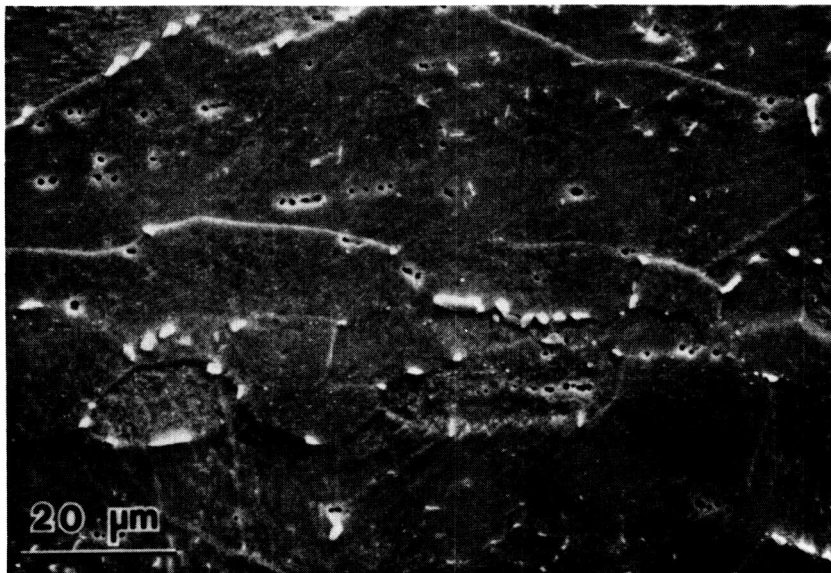
TABLE III. - LATTICE PARAMETER AND STRUCTURE OF POSSIBLE PRECIPITATE PHASES
[Storms, 1967.]

Phase		a ₀ , nm
Nb ₂ C	hcp	0.313 ; c = 0.497
NbO	cubic	.421
NbC	fcc (B1)	.447±
ZrN	fcc (B1)	.456
ZrO	fcc (B1)	.462
ZrC	fcc (B1)	.470±
ZrO ₂	fcc (C1)	.509

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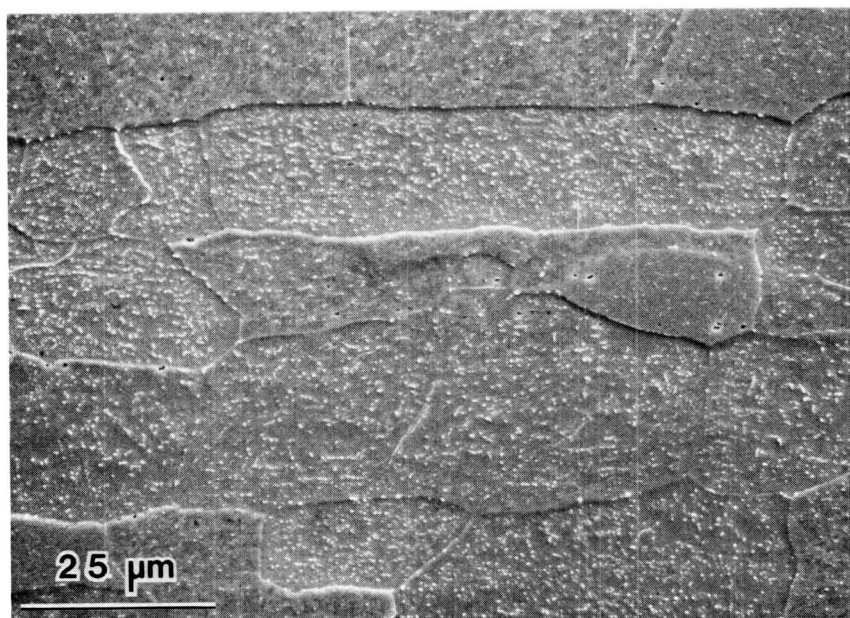


(a) AS-ROLLED SHEET.
← ROLLING DIRECTION →

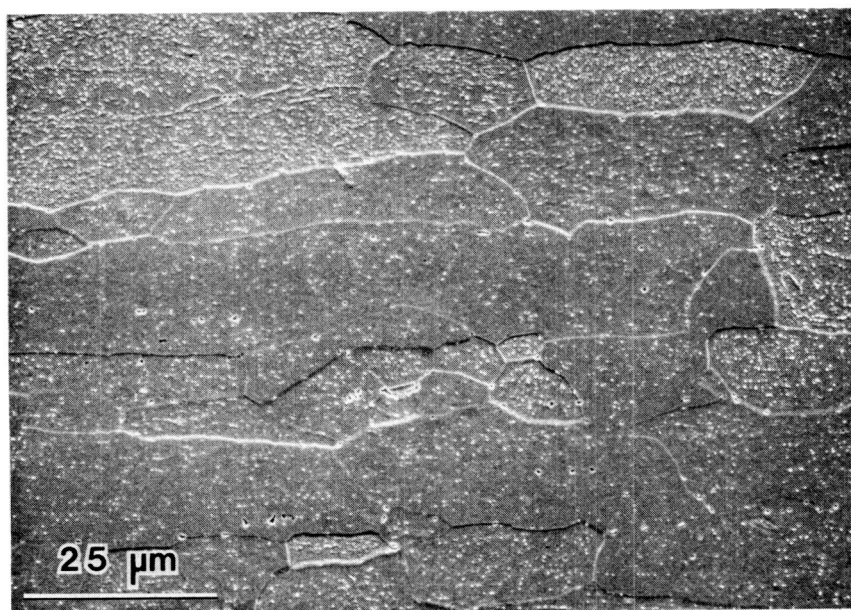


(b) ANNEALED CONDITION.

FIGURE 1. - MICROSTRUCTURE OF Nb-Zr-C ALLOY.



(a) ANNEALED, AGED 1000 HR AT 1350 K, THEN CREPT 770 HR AT 1350 K TO 1.2% STRAIN.



(b) ANNEALED, THEN CREPT 3840 HR AT 1350 K TO 3.3% STRAIN.

FIGURE 2. - MICROSTRUCTURE OF Nb-Zr-C ALLOY AFTER CREEP TESTING.

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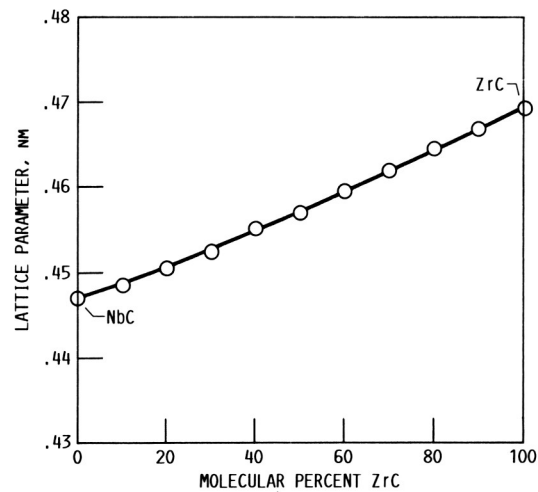


FIGURE 3. - LATTICE PARAMETER-COMPOSITION CURVE
FOR NbC-ZrC (FROM NORTON AND MOWRY, 1949).

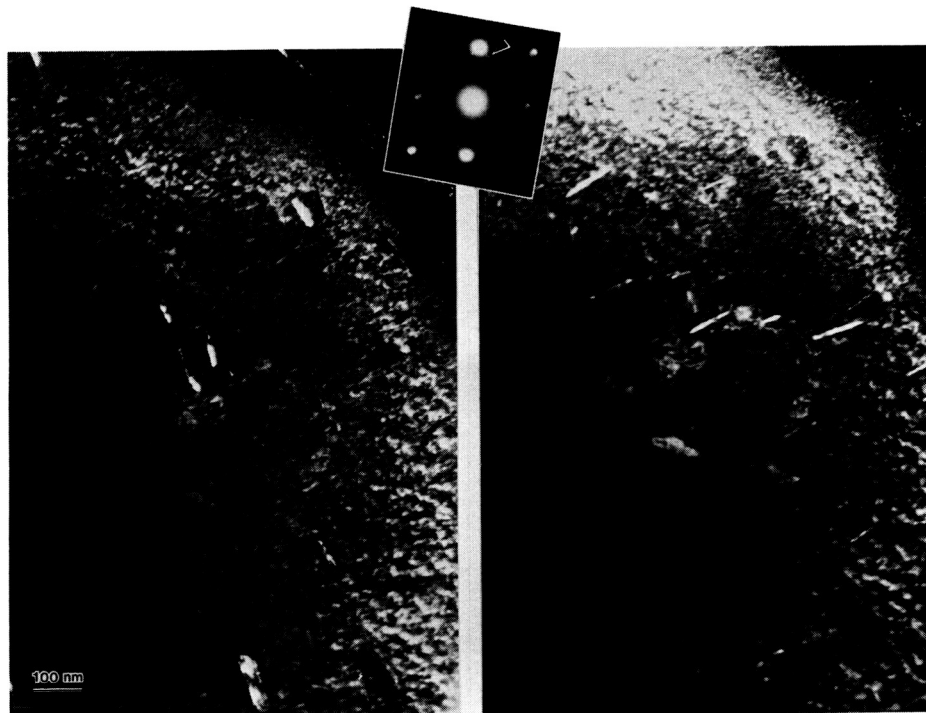


FIGURE 4. - DARK FIELD MICROSTRUCTURE OF Nb-Zr-C ALLOY AFTER 25 HR EXPOSURE AT 1350 K SHOWING
TWO PERPENDICULAR VARIANTS OF A PRECIPITATE PHASE.

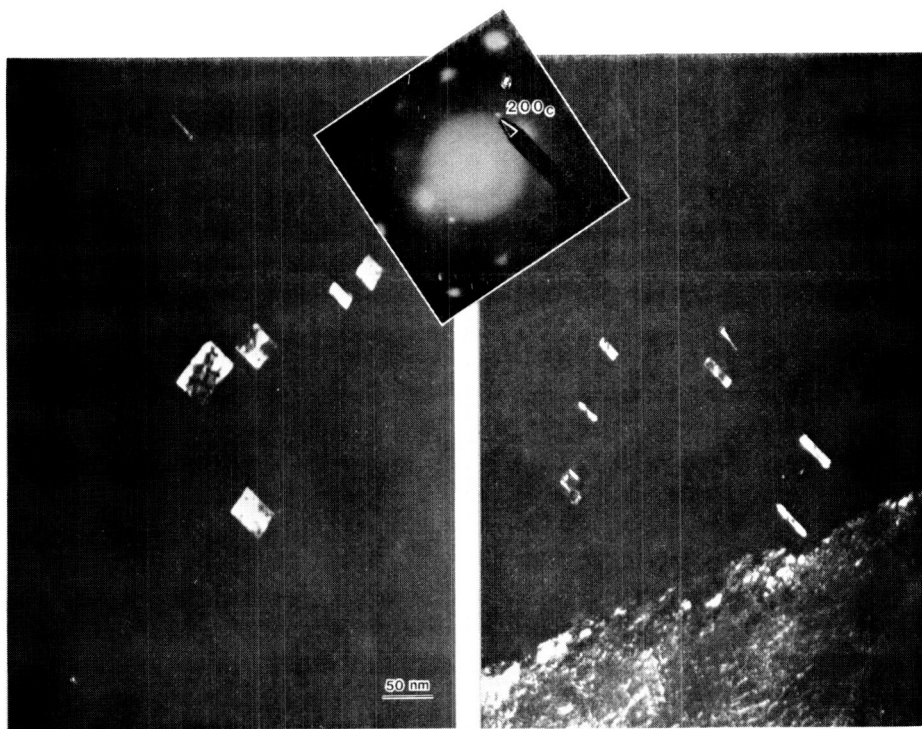


FIGURE 5. - DARK FIELD MICROSTRUCTURE OF Nb-Zr-C ALLOY AFTER BEING ANNEALED, THEN CREEP TESTED AT 1400 K AND 40 MPa FOR 648 HR TO 3.6% STRAIN. TWO PERPENDICULAR VARIANTS ARE SHOWN.

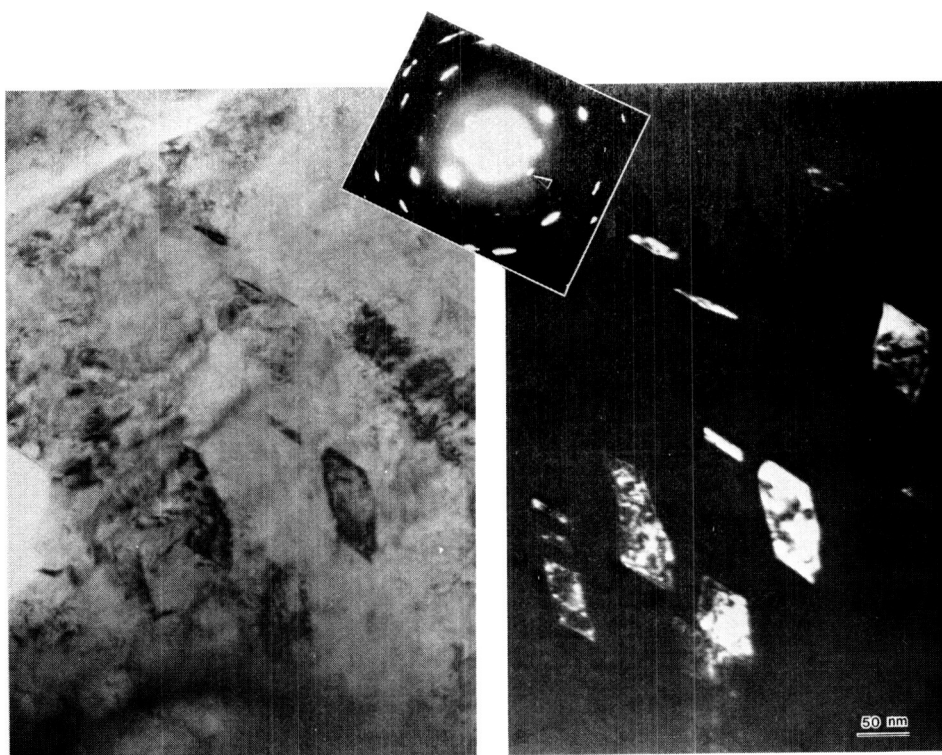


FIGURE 6. - BRIGHT AND DARK FIELD MICROSTRUCTURES OF Nb-Zr-C ALLOY, AS IN FIGURE 5.

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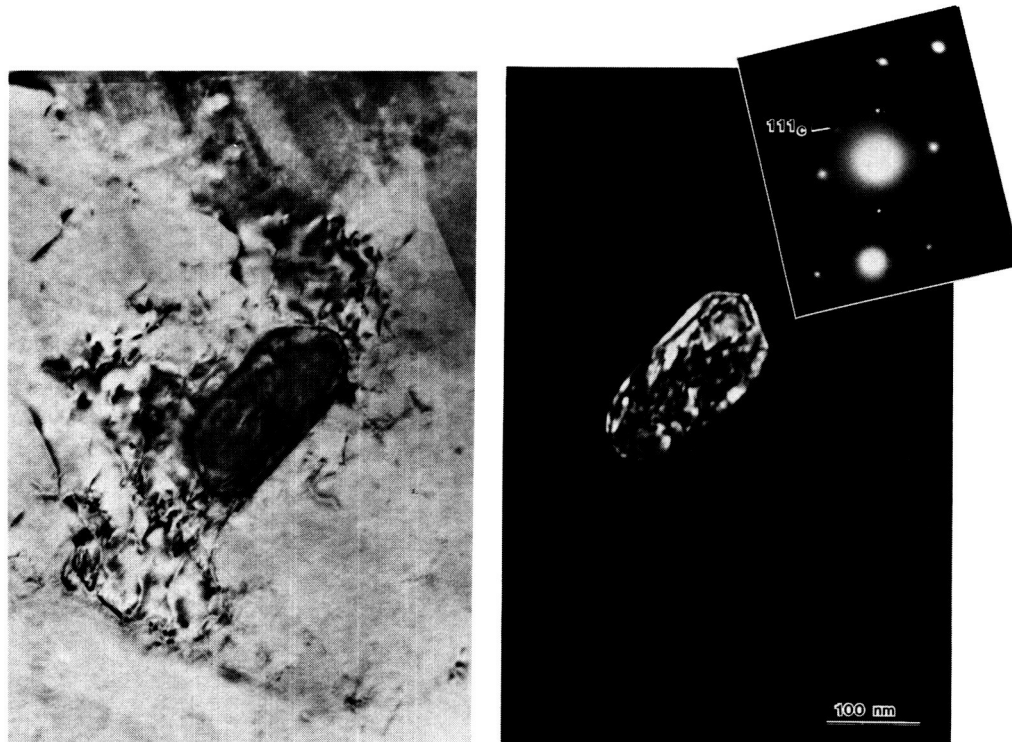


FIGURE 7. - BRIGHT AND DARK FIELD MICROSTRUCTURES OF Nb-Zr-C ALLOY AFTER BEING ANNEALED, THEN CREEP TESTED AT 1350 K AND 40 MPa FOR 4772 HR TO 1.6% STRAIN.

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16. Abstract A niobium alloy with 1 percent zirconium and 0.063 percent carbon by weight was investigated in the as-rolled and annealed conditions, and after high-temperature (1350 and 1400 K) exposure with and without an applied stress. In the as-rolled and in the annealed conditions, large metastable carbides were observed in addition to a regular distribution of small particles. During the high-temperature exposure, the majority of the large carbides were dissolved and a more stable carbide phase formed. This finely dispersed phase had a composition determined to be ~70 percent ZrC and ~30 percent NbC and showed some evidence of an orientation relationship with the matrix. The precipitates appeared to coarsen slightly after ~5000 hr exposure at temperatures between 1350 and 1400 K. This same high-temperature exposure in the presence of an applied stress resulted in a decrease in the size and in the interparticle spacing of the stable precipitates. However, the composition of the precipitate phase and its ability to pin dislocations were not affected by the temperature or stress conditions.					
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